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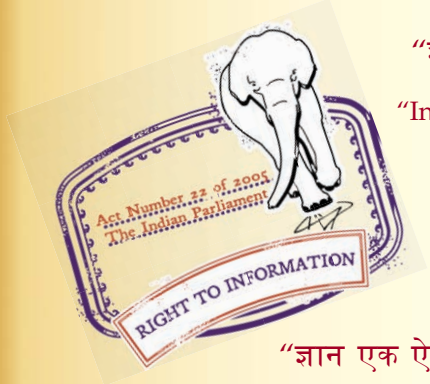
IS 7359 (1992): 1-Chloroanthraquinone, Technical [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]



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"Knowledge is such a treasure which cannot be stolen"

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भारतीय मानक

१-क्लोरोऐन्थ्राक्विनान, तकनीकी – विशिष्टि

(पहला पुनरीक्षण)

Indian Standard

1-CHLOROANTHRAQUINONE, TECHNICAL — SPECIFICATION

(First Revision)

UDC 668·812 : 547·673·2

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

September 1992

Price Group 3

AMENDMENT NO. 1 MARCH 2002
TO
IS 7359 : 1992 1-CHLOROANTHRAQUINONE, TECHNICAL — SPECIFICATION

(First Revision)

(*Foreword, Structural Formula*) — Insert the following below the structural formula:

'(CAS No. 82-44-0)'.

(*Page* 4, *line* 2, *first word*) — Substitute 'optical' *for* 'optional'

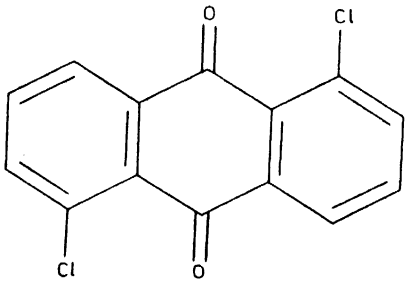
(PCD 11)

Reprography Unit, BIS, New Delhi, India

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standaads, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Divison Council.

1-Chloroanthraquinone ($C_{14}H_7O_2Cl$) is an important intermediate used in the manufacture of vat dyes. It is represented by the following structural formula:



1-CHLOROANTHRAQUINONE

(Molecular Mass 242·65)

This standard was first published in 1974. The committee responsible for the preparation of this standard decided to update it in light of experience gained. In this version, the requirements for assay and the impurities such as 1,5-dichloroanthraquinone and 1,8-dichloroanthraquinone have been stipulated. The requirement for melting point has been modified and oxygen flask method has been introduced for determination of organically bound chlorine.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values *(revised)*'. The number of significant places retained in the rounded off valve should be the same as that of the specified value in this standard.

Indian Standard

1-CHLOROANTHRAQUINONE, TECHNICAL — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and tests for 1-chloroanthraquinone, technical.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
915 : 1975	One-mark volumetric flasks (*first revision*)
1070 : 1992	Reagent grade water — Specification (*third revision*)
1839 : 1961	Toluene, reagent grade
2552 : 1989	Steel drums (galvanized and ungalvanized) (*third revision*)
5299 : 1969	Methods of sampling and tests for dye intermediates

3 REQUIREMENTS

3.1 Description

The material shall be in the form of light yellow powder and shall be free from visible impurities.

3.2 The material shall also comply with the requirements given in Table 1.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in steel drums (*see* IS : 2552 : 1989) lined with suitable polyethylene film or as agreed to between the purchaser and the supplier.

4.2 Marking

4.2.1 Each container shall be securely closed and shall bear legibly and indelibly the following information:

a) Name of the material;
b) Indication of the source of manufacture;
c) Batch number, if any;
d) Net mass of material; and
e) Month and year of manufacture.

4.2.2 Each container shall, in addition, bear the minimum cautionary notice worded as under:

"DANGER! HAZARDOUS, SOLID AND VAPOUR RAPIDLY ABSORBED THROUGH SKIN".

4.2.3 The containers may also be marked with the Standard Mark.

Table 1 Requirements for 1-Chloroanthraquinone, Technical

(*Clauses* 3.2, 5.3.1 *and* 6.1)

Sl No.	Characteristic	Requirement	Methods of Test, Ref to
(1)	(2)	(3)	(4)
i)	Melting range	Shall melt within the range of 158° to 160°C	3 of IS 5299 : 1969
ii)	Moisture content, percent by mass, *Max*	0.5	9.3 of IS 5299 : 1969
iii)	Sulphated ash content, percent by mass, *Max*	0.5	11.2 of IS 5299 : 1969
iv)	Chlorine content, percent by mass	14.0 to 15.0	Annex A
v)	Matter insoluble in hot toluene, percent by mass, *Max*	1.0	Annex B
vi)	Assay, percent by mass, *Min*	96	Annex C, D
vii)	Impurities: a) 1,5-dichloroanthraquinone, and b) 1,8-dichloroanthraquinone, percent by mass, *Max*	3	Annex C, D

5 SAMPLING

5.1 Representative samples of the material shall be drawn as prescribed in 3 of IS 5299 : 1969.

5.2 Number of Tests

5.2.1 Test for the determination of chlorine, assay and impurities shall be conducted on each of the individual samples.

5.2.2 Tests for the determination of the remaining characteristics, namely, melting point, moisture content, sulphated ash and matter insoluble in hot toluene shall be conducted on the composite sample.

5.3 Criteria for Conformity

5.3.1 For declaring the conformity of the lot to the requirements of this specification, all test results on individual sample as well as composite sample shall satisfy the relevant requirements given in Table 1.

6 TEST METHODS

6.1 Tests shall be carried out according to the methods prescribed in Annexes and IS 5299 : 1969, as indicated in col 4 of Table 1.

6.2 Quality of Reagents — Unless specified otherwise, 'pure chemicals' and distilled water (*see* IS 1070 : 1992) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[*Table* 1, *Item* (iv)]

DETERMINATION OF CHLORINE BY OXYGEN FLASK METHOD

A-1 APPARATUS

A-1.1 Whatman Filter Paper No. 44 — sheet for the preparation of paper packet.

A-1.2 Ignition Flask

Place 25 ml distilled water in a clean 500 ml quickfit flask, add 5 ml of 2 N sodium hydroxide solution and 0·5 ml 100 volume hydrogen peroxide. Pass oxygen gas from O_2 gas cylinder for 5 minutes to replace air from the flask and stopper immediately. Cover the flask with wire gauge for safety.

A-1.3 Wire Gauze

A-1.4 Platinum Wire

A-1.5 Beaker — 500 ml capacity.

A-1.6 Gooch — crucible.

A-1.7 Oven — capable of maintaining temperature at $100^{\circ} \pm 5^{\circ}C$.

A-2 REAGENTS

A-2.1 Sodium Hydroxide Solutions — 2 N.

A-2.2 Hydrogen Peroxide — 100 volume.

A-2.3 Sulphuric Acid — 2 N.

A-2.4 Sodium Bisulphite — 2 percent (*m/v*).

A-2.5 Nitric Acid — 60 percent (*m/v*).

A-2.6 Silver Nitrate Solution — N/10 (*m/v*).

A-3 PROCEDURE

A-3.1 Weigh accurately by difference method about 0.04 g to 0.05 g of sample (previously sieved and dried) in a paper packet of Whatman Filter Paper No. 44 sheet previously folded using a small aluminium scoop. Place the paper packet in the gauze cup attached to platinum wire of the stopper, keeping the end of paper strip slightly projected.

A-3.2 Ignite the tip of the paper packet. When it just reaches the packet, immediately insert it into the flask and hold the stopper firmly. Cover the flask with wire gauze for safety. After ignition is over, tilt the flask upside down so that the solution seals the flask from inside. Keep the flask rightdown for 15 minutes. Intermittently shake the flask vigorously so as to ensure complete absorption of fumes. Remove the stopper, wash platinum wire and gauze quantitatively with few ml of distilled water into the flask. The solution is ready for estimation.

A-3.3 Chlorine Estimation

Transfer the solution quantitatively into 500 ml beaker and boil for 5 minutes to remove peroxide, cool and neutralize with 2 N sulphuric acid (normally 5 ml is required). Then add 10 ml of 2 percent sodium bisulphite solution, 10 ml of 60 percent nitric acid. Boil to expel SO_2 gas. Cool and filter the solution through Buchner funnel dressed with double filter paper No. 1

Transfer the filtered solution quantitatively to 500 ml beaker and add 10 ml of N/10 $AgNO_3$. Boil to coagulate the precipitates. Cool and filter through previously weighed Gooch crucible. Wash the precipitates to make it acid and chloride free. Then dry at $100^{\circ} \pm 5^{\circ}C$ to constant mass.

A-4 CALCULATION

A-4.1 Chlorine content, percent by mass $= \dfrac{M \times 24{\cdot}74}{M_1}$

where

M_1 = mass of sample taken, and

M = mass of precipitate (AgCl).

ANNEX B

[*Table* 1, *Item* (v)]

DETERMINATION OF MATTER INSOLUBLE IN HOT TOLUENE

B-1 APPARATUS

B-1.1 Conical Flask — 500 ml.

B-1.2 Sintered Glass Crucible — G 4, two numbers.

B-1.3 Reflux Condenser

B-2 REAGENT

B-2.1 Toluene — *See* IS 1839 : 1961.

B-3 PROCEDURE

Weigh accurately about 1 g of the sample in a 500 ml conical flask and add about 150 ml of toluene to the flask. Reflux for about 30 minutes using a water condenser. Filter on a tared sintered glass crucible. Wash the conical flask, two to three times with hot toluene and transfer the washings also to the crucible. Wash the crucible again two to three times with hot toluene. Dry the sintered glass crucible in an oven at 100° to 110°C for four hours. Cool the crucible to room temperature in a desiccator and weigh. The increase in the mass of the crucible is the mass of insolubles.

B-4 CALCULATION

$$\text{Matter insoluble in toluene, percent by mass} = \frac{M_1}{M_2} \times 100$$

where

M_1 = mass in g of insolubles in toluene, and

M_2 = mass in g of material taken.

ANNEX C

[*Table* 1, *Items* (vi) and (vii)]

DETERMINATION OF ASSAY AND IMPURITIES

C-1 GENERAL

Assay and impurities like 1,5-dichloroanthraquinone and 1,8-dichloroanthraquinone are estimated by infra-red spectrophotometric method.

C-2 REAGENTS

C-2.1 1-Chloroanthraquinone — pure (100 percent, on dry basis).

C-2.2 1,5-Dichloroanthraquinone — pure (100 percent, on dry basis).

C-2.3 1,8-Dichloroanthraquinone — pure (100 percent, on dry basis).

C-2.4 1,4-Dioxane — dry.

C-3 APPARATUS

C-3.1 Infra-red Spectrophotometer — Any suitable instrument.

C-3.2 Cells — Two fixed cells for liquid with NaCl window and thickness 0·5 mm.

C-3.3 One-Mark Volumetric Flasks — *See* IS 915 : 1975.

C-4 PROCEDURE

C-4.1 Preparation of Sample Solution

Weigh accurately about 150 mg of dry pulverized and sieved (100 mesh) sample in a 25 ml volumetric flask. Add about 10 to 15 ml of dry 1,4-dioxane and heat to boil on a hot plate in order to dissolve the sample completely. Cool to room temperature. Dilute up to the mark and shake well.

C-4.2 Preparation of Standard Solution

Weigh accurately about 150 mg of pure 1-chloroanthraquinone (**C-2.1**) in a 25 ml volumetric flask. Add accurately weighed about 3 mg each of 1,5-dichloroanthraquinone (**C-2.2**) and 1,8-dichloroanthraquinone (**C-2.3**). Add about 10 to 15 ml of dry dioxane (**C-2.4**) and heat to boil on a hot plate to dissolve the contents. Cool to room temperature. Dilute to the mark and shake well.

C-4.3 Fill the reference cell and sample cell of 0.5 mm path length with dry dioxane and adjust the instrument for the 100 percent transmittance. Fill the

sample cell with standard solution and read the optional density at various wavelengths which corresponds to different components as under:

Component	*Wave length*
1-chloroanthraquinone	964 cm^{-1}
1,8-dichloroanthraquinone	733.3 cm^{-1}
1-chloroanthraquinone plus 1,5-dichloroanthraquinone	709.3 cm^{-1}

Read the optical density of the standard solution at the above wave lengths.

Similarly run the instrument with sample solution and note the optical densities at the above wavelength.

C-5 CALCULATION

C-5.1 1-chloroanthraquinone, percent by mass $= \frac{A \times B \times C}{D \times M}$ Say, X_1

where

A = optical density of sample at 964 cm^{-1};

B = percent content of 1-chloroanthraquinone in standard solution;

C = mass of pure 1-chloroanthraquinone;

D = optical density of standard solution at 964 cm^{-1}; and

M = mass of sample taken.

C-5.2 1,8 dichloroanthraquinone, percent by mass $= \frac{A_1 \times B_1 \times C_1}{D_1 \times M_1}$

where

A_1 = optical density of sample at 733.3 cm^{-1};

B_1 = percent content of 1,8-dichloroanthraquinone in standard solution;

C_1 = mass of pure 1,8-dichloroanthraquinone;

D_1 = optical density of standard solution at 733.3 cm^{-1}; and

M_1 = mass of sample taken.

C-5.3

1,5-dichloroanthraquinone plus 1-chloroanthraquinone, percent by mass $= \frac{A_2 \times B_2 \times C_2}{D_2 \times M_2}$ = Say, Y_1

where

A_2 = optical density of sample at 709.3 cm^{-1};

B_2 = percent content of 1-chloroanthraquinone plus 1,5-dichloroanthraquinone in standard solution;

C_2 = mass of pure isomers;

D_2 = optical density of standard solution at 709.3 cm^{-1}; and

M_2 = mass of sample taken.

C-5.4 1,5-dichloroanthraquinone content, percent by mass $= Y_1 - X_1$

where

Y_1 = as calculated in **C-5.3**, and

X_1 = as calculated in **C-5.1**.

ANNEX D

[*Table* 1, *Items* (vi) *and* (vii)]

ASSAY AND DETERMINATION OF IMPURITIES (ALTERNATE METHOD)

D-1 GENERAL

Assay and impurities like 1,5-dichloroanthraquinone and 1,8-Dichloroanthraquinone are estimated by High Pressure Liquid Chromatographic method.

D-2 REAGENTS

D-2.1 1-Chloroanthraquinone — Pure (100 percent, on dry basis).

D-2.2 1,5-Dichloroanthraquinone — Pure (100 percent, on dry basis).

D-2.3 1,8-Dichloroanthraquinone — Pure (100 percent, on dry basis).

D-2.4 Acetonitrile — Spectroscopy grade.

D-3 APPARATUS

D-3.1 High Pressure Liquid Chromatograph

Any suitable chromatograph with a reverse phase column. A set of typical parameters of HPLC is given below:

i) *Column* — μ Bondapak C18

ii) *Temperature* — 25°C

iii) *Wavelength* — 254 nm

iv) *Flow Rate* — 1.4 ml/Min

v) *Attenuator* — 0.1

vi) *Chart Speed* — 10 mm/Min

vii) *Load on Column* — 5 μl

viii) *Solvent System* — Acetonitrile : Water : : 60 : 40

D-3.2 'A' Grade Volumetric Flask

D-4 PROCEDURE

D-4.1 Preparation of Sample Solution

Weigh accurately 0.025 g of well mixed (previously ground dried) sample and transfer into 100 ml volumetric flask using 80 ml of acetonitrile (**D-2.4**) and heat to boil on hot plate to dissolve the sample. Cool to room temperature. Dilute to mark using acetonitrile and mix well. Call this solution S.

Pipette 5.0 ml of solution S into 100 ml standard volumetric flask and dilute to 100 ml mark using acetonitrile. Mix well. Call this solution S1.

D-4.2 Preparation of Standard Solutions

1) **1-Chloroanthraquinone** — Pure

Weigh accurately 0.025 g of pure 1-Chloroanthraquinone previously dried and transfer into 100 ml standard volumetric flask using 80 ml of acetonitrile (**D-2.4**). Heat to boil on hot plate to dissolve. Cool at room temperature and make up to mark using acetonitrile. Mix well. Call this solution A.

Pipette 5 ml of solution A into 100 ml standard volumetric flask and dilute to 100 ml mark using acetonitrile. Mix well. Call this solution A1.

2) **1,8-Dichloroanthraquinone** — Pure

Weigh accurately 0.05 g of pure 1,8-Dichloroanthraquinone previously dried and transfer to 100 ml standard volumetric flask using 80 ml of acetonnitrile. Proceed as above to prepare solution B and B1.

3) **1,5-Dichloroanthraquinone** — Pure

Weigh accurately 0·05 g of pure 1,5-Dichloroanthraquinone previously dried. Proceed in the same way to prepare solution C and C_1.

D-4.3 When steady base line is obtained, inject 5 μl solution of each standard from solutions A1, B1 and C1. Also inject 5 μl of sample solution S1.

Measure the peak height and base width of the peak and calculate.

D-4.4 Calculation

$$\text{1-chloroanthraquinone, percent by mass} = \frac{\text{Concentration of standard} \times \text{Area of Peak in Sample} \times \text{Strength of Standard}}{\text{Concentration of sample} \times \text{Area of peak of Standard}}$$

1,5-Dichloroanthraquinone, percent by mass = As above

1,8-Dichloroanthraquinone, percent by mass = As above

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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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Printed at Printrade, New Delhi, India